

A workshop to promote the use of high energy x-ray diffraction experiments and detailed computational analyses for understanding multiscale phenomena in crystalline materials

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**On the Use of HEXDM Data for Characterizing and Validating
a Mesoscale Model of Dislocation Plasticity**

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The kinematic ingredients/state variables of a mesoscale pde model of dislocation mechanics and plasticity will be discussed to facilitate correlation with HEXDM measurements. The fundamental statistical mechanical definition of a mesoscale entropy function (that can, e.g., greatly aid in defining constitutive response for a back stress tensor) will be discussed and the possibility of parametrizing it from many, many snapshots of HEXDM data will be explored.

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Multiscale Modeling of States in Granular Media

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In this talk, I will present recent work using powerful characterization tools, such as 3DXRCT and 3DXRD, to construct predictive multiscale models for granular materials. Particle kinematics have been used to construct multiscale models and have been shown to represent material behavior at the continuum level very well. However, inference of inter-particle forces in non-birefringent particulate materials remains as one of the ultimate frontiers in the field. New tools using 3DXRD have allowed direct measurement of average elastic strains in particulate media under static macroscopic loading. We will show a new method that will enable, for the first time, the inference of inter-particle forces by using data extracted from 3DXRD. These new multiscale tools will enable the new generation of models that will construct granular material state directly from micro-structural process, rather than from limited phenomenology.

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Predicting Internal Stresses with a Consistent Thermodynamic Theory of Crystals Containing Defects

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(Co Authors: Jason Mayeur, David L. McDowell)

An internal state variable theory of micropolar elasto-viscoplasticity is developed based upon the physics associated with dislocations and disclinations. Elastic-plastic kinematics are modified to include an additional rotational degree of freedom from which non-symmetric elastic and plastic strains and curvatures are defined. This is essential to have kinematics with consistent degrees of freedom to accommodate the defects introduced into the theory. Dislocations and disclinations can then be easily identified in terms of the incompatibilities associated with the elastic deformation and elastic curvature. The state variables introduced are the non-symmetric internal elastic strain and elastic curvature resulting from the presence of the dislocations and disclinations, as well as scalar measure of the elastic strain field associated with the statistically stored dislocations. The conjugate thermodynamic internal micro-stress and micro-moment are required to satisfy micro linear and angular momentum balances, while the macro stress (the derivative of the free energy with the respect to the macro elastic strain) satisfies standard linear and angular (symmetry of stress tensor) momentum balance laws. Expressions for the plastic velocity gradient and plastic curvature are proposed as well as an equation describing the evolution of the statistically stored dislocation density. The resulting expression describing the dissipation associated with the micro and macro stress fields follows naturally as a result of the second law, and the ramifications these restrictions on localized deformation is discussed. This model has been implemented into the finite element code ABAQUS and comparisons are made with dislocation dynamic (DD) simulations, where it is shown that the torsion –curvature more accurately compares with DD predictions, than theories containing the curl of the elastic deformation gradient (representing geometrically necessary dislocations) and maintaining symmetry of the stress.

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Elastic/Plastic Interface Interactions in Composites: 3D Micro-Laue Diffraction and Modeling

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Nearly perfect fiber/matrix composites formed from directionally solidified eutectic provide model systems of small-scale and interface behavior. To understand the small-scale mechanical behavior of microfibers/lamellae in composite materials, it is critical to characterize their defect distributions at appropriate length scales and compare it with modeling results. A characterization technique appropriate for these samples, based on polychromatic and scanned monochromatic microdiffraction method using Differential Aperture X-ray Microscopy (DAXM) is described. Modeling of the composites respond to different external loading is compared to the experimental ones. A micromechanical model that assumes addition of thermal residual strains and indentation-induced strain fields leads to a fair prediction of the change of measured lattice strains in both fiber and matrix phases. The 3D DAXM measurements also call for a more detailed finite element simulation with explicit description of the 3D microstructure.

Research supported by the Materials Sciences and Engineering Division, Office of Basic Energy Sciences, U.S. Department of Energy.

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Finite Element Modeling of High Energy X-ray Diffraction Microscopy Results

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(Co-Authors, J. Bernier, J. Edmiston & M. Miller)

High energy X-ray diffraction microscopy experiments provide detailed information from bulk grains, including lattice orientations, lattice stretches, and grain centers of mass. These quantities can be collected during in situ deformation experiments and used to quantitatively examine mechanical properties of materials. These data also allow for three-dimensional microstructure generation, and therefore direct comparison with numerical simulations of deformation of as-measured microstructures. We will present and compare experimental and simulation results for a continuously loaded α titanium alloy.

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

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In-Situ Measurement of Lattice Strain in Al-Li Alloys

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(Co-Authors: K. Halm, M. Obstalecki, R. Storer, W. Tayon, U. Lienert & P. Kenesei)

High Energy Diffraction Microscopy provides the capability to query crystallographic orientation and elastic strain within the sample volume of a polycrystalline material. Combined with in situ loading, data for the elasto-plastic transition on a grain-by-grain basis is accessible. That this data is for grains in a constrained environment lends considerable value in assessing material performance. Models for crystal plasticity coordinated with experiments provide a cross-check for -- as well as advance the interpretation of -- experimental results. In this presentation, studies of Al-Li alloys at the beamline 1-ID of the Advanced Photon Source of Argonne National Laboratory will be reviewed. Techniques that aid in the examination of microstructure for a production alloy, such as use of the conical slit cell, will be illustrated. We then draw implications for models of plasticity and damage evolution.

This work is supported by the NASA Marshall Flight Center through grant NNX09AN21G and Dept. of Energy grant DE-FG36-05GO15049.

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High Energy Diffraction Microscopy as a Tool for high Pressure Research

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(Co-Authors: N. Barton & D. Boyce)

The marriage of high energy diffraction microscopy (HEDM) and the diamond-anvil cell has the potential to revolutionize experiments in high pressure physics. The 3-dimensional nature of the measurements alleviate the ambiguity in the non-hydrostatic component of the applied stress, enable determination of parent/product orientation relationships and preferred variant selection for phase transformations and/or twinning, and provide enough data to perform detailed structure refinement even for heavily deformed materials. The unit cell volume can also be determined to very high precision, which is critical to accurate equation of state (EOS) measurements. The fundamental aspects of the HP-HEDM experiment are illustrated using recent measurements from APS 1-ID that show the $\alpha \rightarrow \epsilon$ phase transformation in iron. Two crystallographic mechanisms have been proposed in the literature for this well studied system; however, until application of the HEDM method there have been no measurements that unambiguously demonstrate which is correct. The HEDM data clearly illustrate the Burgers mechanism for the phase transformation under quasi-static deformation, with variant selection correlated to the magnitude of the stress deviator. Such precise characterization of phase transformation mechanisms is simply not possible via other methods. We conclude with a brief discussion of the outlook for HP-HEDM experiments in the near and long term.

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

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HEXRD: A modular software suite for analysis of high-energy X-ray diffraction data

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(Co-Authors: N. Barton & D. Boyce)

(Saturday Lecture) In this workshop we present HEXRD, a fully open source, modular, Python-based software suite for the analysis of high-energy diffraction images. The top-level functionalities include:

- high-precision flat-panel detector calibration via powder and/or single crystal standards;
- interactive image browsing;
- polar re-binning (i.e. “caking”) and line position extraction for powder diffraction images;
- image segmentation for far-field HEDM oscillation image stacks (i.e. “spot finding”)
- orientation indexing of far-field HEDM spot data; and
- cell refinement for indexed grains.

The object design is presented in the context of the experiment -- both on-line and off-line analysis -- and interface, including compatibility and interoperability with Fable components. This is accompanied by a live demo of the current software package. The informal setting should provide the basis for an interactive discussion on what the needs of the user community are moving forward. This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under Contract DE-AC52-07NA27344.

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Microstructural characterization of activated materials with high energy x-ray diffraction

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(Co-Authors: Sisneros, Mosbrucker & Balogh)

The lifetime of a nuclear fuel pellet assembly is to a large degree determined by the structural integrity of the fuel cladding and thermal conductivity of the ceramic fuel. Both of these are dictated by the microstructure of the material. In service, the extreme thermal gradient drives radial grain growth and void migration which deteriorates thermal conductivity and facilitates transport of fission products. Likewise, radiation damage in the fuel cladding deteriorates the mechanical properties, e.g. reduces the ductility. Thus, being able to monitor the evolution of the microstructure of fuel and cladding materials under conditions approaching those in reactor is necessary in order to make informed decisions concerning lifetime of nuclear fuel assemblies, or to possibly optimize their behavior. High energy x-ray diffraction provides this possibility in some cases. In particular, this talk will present some recent results on microstructural evolution of steel samples after 6 years of service in reactor and will discuss planned measurements on ceramic nuclear fuels under extreme thermal gradients.

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High-Energy X-Ray Diffraction on Material Science/Engineering Systems at NSLS I & II

M. Croft, Rutgers Univ., croft@physics.rutgers.edu

Facets of the superconducting wiggler (SCW) based, high-energy diffraction programs and facilities at NSLS I will be discussed. Applications to fatigue crack growth (and the constraints imposed upon modeling) and to in situ real battery electrochemistry will be briefly discussed. The proposed "HEX" material science/engineering, high-energy, NSLS II beam line will have 20 X the flux, at energies above 150 keV, of the NSLS I facility. The potential of this new beamline to substantially contribute to assembling critical mass in the national 3DXRD/multiscale-modeling will be noted. The other, complementary techniques which will be supported by the HEX beamline will also be noted.

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FE crystal plasticity codes; comparisons with diffraction data

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Twinning plays an important part in the deformation of many metals, particularly those with lower crystal symmetries, for example zirconium, magnesium or titanium and their alloys with an h.c.p. crystal structure. The occurrence of twinning in these materials is due to the fact that while slip can occur easily within the basal plane, it is often not an easy mode when a component of deformation is perpendicular to the basal plane, hence instead twinning may be favored. Twinning is typically considered to occur when a critical resolved shear stress (CRSS) is exceeded, in analogy to that required for slip. However, due to the surface energy term associated with the twin-parent boundary, it is usually considered that nucleation and propagation require a different CRSS. A newly nucleated twin will propagate, extending rapidly to some energetically favorable size, relaxing the stress state in the parent grain compared to that just prior to twin formation. This creates a complex situation where the resistance (i.e. CRSS) and driving force (i.e. stress state within parent) for twin formation change rapidly as the twin nucleates and initially propagates. This process also generates a back-stress within the twin itself. Such a process is heavily influenced by local neighborhood effects. We describe studies investigating the stress states associated with twin formation and propagation, combining FE crystal plasticity models with diffraction measurements.

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Using Crystal-Based Finite Elements Simulations and Diffraction Experiments to Understand the Behavior of Deforming Polycrystals

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(Co-Author: M. Miller)

How a polycrystalline solid deforms and perhaps ultimately fails under applied loads depends on a number of attributes of the crystals that comprise the solid and the interactions between those crystals. Foremost among a crystal's attributes are its elastic and plastic properties, both of which can exhibit significant anisotropy. The constraints imposed by equilibrium and compatibility force crystals within an aggregate to adjust under the influence of their neighbors. Consequently, the fields of stress and deformation over a polycrystal are spatially heterogeneous at the microscopic scale, even for nominally uniform loadings. Coordinated simulation and experiment is a potent approach to sorting out the influence of various attributes of crystals on the properties of polycrystalline materials. In particular, diffraction experiments with in situ loading combined with finite element simulations of virtual polycrystals complement each other with intra- and inter-crystalline information sufficient to observe and interpret the heterogeneous stress and deformation fields in polycrystalline solids. Using these combined capabilities as a single integrated tool, we can examine a number of aspects of the behavior of polycrystalline alloys that remain inadequately understood, but are central to using better materials or better using the materials we have. Some examples are: how the stiffness and strength influence load sharing between phases and between crystals within a phase; how fatigue defects initiate under macroscopic loading that is nominally elastic; and how alloys change phases when subjected to changing temperature or deformation. These behaviors are difficult to understand without the more complete picture of the microstructural state and mechanical loading made possible with using coordinated simulation and experiment at the micromechanical level. In this talk, we present some examples from collaborations among the modelers and experimentalists of our research group that provide a basis for better understanding metallic alloys, such as quantifying the orientational dependence of the crystal stress and the influence of directional strength-to-stiffness on hysteresis loops in cyclic loading. We conclude with a discussion of the current limitations we face, in both experiment and simulations, and suggest some avenues for future work.



Industrial Applications for HEDM/3DXRD

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While traditional two-dimensional analysis of materials has enabled the vast bulk of modern technology, characterizing the three-dimensional microstructure of crystalline materials has important industrial applications. In particular, understanding deformation and failure mechanisms is of significant interest, as these have a direct impact on product function, lifespan, and associated costs. Recently, the maturation of HEDM/3DXRD synchrotron technology has demonstrated significant promise towards solving several engineering problems. While destructive methods using EBSD analysis offer similar 3D data with better spatial resolution, HEDM/3DXRD has the critical advantage of being non-destructive. Efforts towards in-situ testing at stresses and temperatures of interest will therefore provide the industrial community with the most benefit.

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Validation of lattice stress from diffraction measurements in polycrystalline solids under elastic-plastic deformation using stress evolution pattern analysis

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The synergic approach of combining the state-of-the-art experiment and simulation tools for investigating the behavior of polycrystalline materials becomes more effective as advanced X-ray diffraction measurement techniques become available. The lattice strain evolution in loading and transverse directions measured from a diffraction experiment was confirmed accurate when compared with a simulation tool such as finite element analysis [1]. As strain pole figures are obtained from X-ray diffraction experiment, full stress tensors from the measurements were also compared with those from a simulation tool and the reliability of the measured data was verified [2]. The integrity of the measured lattice stress evolution data of a polycrystal aggregate under elastic-plastic deformation can be identified by comparing not only the stress levels between the experiment and simulation but also the pattern of lattice stress direction change. The tendency that the lattice stress tends to move closer to the single crystal yield vertex during plastic flow can be validated by investigating the proximity of the lattice stress to the single crystal yield surface vertex analysis. A direct comparison of full lattice stress tensor evolution measured from a single crystal between the diffraction experiment and simulation is difficult since the initial and boundary conditions at the single crystal level needed for the simulation are not readily available from the experiment [3]. A study on comparing the lattice stress evolution pattern between the measured and simulated data are being conducted to ensure the same characteristics can be found from both approaches.

[1] T.-S. Han and P.R. Dawson. Lattice strain partitioning in a two-phase alloy and its redistribution upon yielding. *Mat. Sci. Eng. A-Struct.*, 405:18–33, 2005.

[2] M.P. Miller, J.-S. Park, P.R. Dawson, and T.-S. Han. Measuring and modeling distributions of stress state in deforming polycrystals. *Acta Mater.*, 56:3927–3939, 2008.

[3] P. Hedström, T.-S. Han, U. Lienert, J. Almer, and M. Odén. Load partitioning between single bulk grains in a two-phase duplex stainless steel during tensile loading. *Acta Mater.*, 58:734–744, 2010.

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DIGIgrain: A Software for X-ray Diffraction Image Analysis and Data Reduction

P. Kenesei, Argonne Nat. Lab., APS, kenesei@aps.anl.gov

(Co-Authors: A. Borbely, U. Lienert)

(Saturday Lecture) DIGIgrain has been developed for enabling the best available physical analysis on a diffraction scan series including all the usual measurement errors and distortions. After the data correction a sophisticated three-dimensional blob/spot/peak searching algorithm is used to identify the diffracted spots similarly as one would do it by natural intelligence and to calculate the necessary descriptors for further analysis, e.g. filtering, indexing, strain tensor fitting, or store these reduced dataset instead of the huge amount of original dataset. The program is written in C and runs parallel in a multiprocessor environment. I will present a brief overview about the algorithms, and some example about the results in different applications.

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The Combined Use of 3D X-Ray Microscopy and Theoretical Modeling to Probe Mesoscale Deformation

B. Larson, Oak Ridge Nat. Lab., larsonbc@ornl.gov

Because of the complex and collective nature of deformation, combining 3D x-ray microscopy and theoretical modeling is critical not only to obtaining a fundamental understanding deformation on mesoscopic length scales, but also for obtaining a full characterization of the nature and complexity of the deformation state of applied and industrial materials. Techniques for handling the single dislocation microscale regime of deformation are relatively successful, as are continuum techniques for handling deformation in materials when the homogeneous macroscale regime is a good approximation. However, combining insight from these two extremes provides a relatively sparse amount of insight into deformation in polycrystalline materials. In this presentation, the combination of submicron resolution 3D x-ray microscopy and discrete dislocation dynamics simulations to probe understanding of the initial stage of deformation on mesoscopic length scales in a single crystal will be discussed and used as an illustration to discuss the value of combining experiment and simulations in mesoscale materials investigations. Research supported by the Department of Energy, Office of Science, Materials Science and Engineering Division and by the Scientific User Facilities Division.

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Synchrotron Studies of 3D Grain Growth Kinetics

E. Lauridsen, Risø /DTU, emla@risoe.dtu.dk

In this presentation we will give examples of 3D imaging of evolving microstructures during grain growth in ceramic and metallic materials. The experiments are conducted at the European Synchrotron Radiation Facility, France, using either phase contrast tomography, diffraction contrast tomography or a combination of both. Emphasis will be on the current experimental capabilities and on the coupling of time-resolved 3D experimental data to large-scale 3D simulations of grain growth kinetics.

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High-energy Diffraction Microscopy at the APS 1-ID Beamline

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(Co-Authors: Peter Kenesei, Robert Suter, Matthew Miller, Wolfgang Pantleon)

The 1-ID beamline at the Advanced Photon Source is dedicated to high-energy diffraction. The high-energy diffraction microscopy (HEDM) program aims at the structural in-situ characterization of polycrystalline bulk materials subjected to external loads on the grain and sub-grain scale. Distinct techniques have been implemented based on the 3DXRD principle, i.e. the simultaneous observation of diffraction from many grains by area detectors. The temporal, real-, and reciprocal-space resolutions will be discussed and illustrated by case studies that range from an individual dislocation in a sub-grain to orientation mapping of millimeter sized samples. Conclusions in view to future instrumental and software developments will be presented.

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Studying Fatigue Damage in Polycrystalline Materials by Combined Use of Phase and Diffraction Contrast Tomography

W. Ludwig, Euro. Synch. Rad. Facil., wolfgang.ludwig@esrf.fr

The bulk propagation of fatigue cracks can be characterized by 'phase contrast tomography' (PCT). When combined with a nondestructive 3D grain mapping technique like 'diffraction contrast tomography' (DCT), details of the crystallographic crack propagation, observed during early stages of fatigue damage can be analyzed. The potential and limitations of this combined characterization approach are discussed.

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"Low Energy" Diffraction Microscopy and an Iterative Algorithm for Diffraction Data Analysis

W. Ludwig, Euro. Synch. Rad. Facil., wolfgang.ludwig@esrf.fr

(Saturday Lecture) A modified 3DXRD acquisition geometry, optimized for low X-ray energies, and the introduction of beam structuring elements in the incoming and/or diffracted beam are discussed. The modified acquisition procedure enables determination of the local directions of the diffracted radiation. An iterative algorithm, aiming at the reconstruction of all nine components of the distortion tensor from monochromatic beam diffraction data is presented.

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**Investigating Electromigration Using X-Ray Microbeam Diffraction
Coupled with Modeling and Simulation**

A. Maniatty, Rensselaer Poly. Inst., maniat@rpi.edu

Electromigration is a mass transport process that occurs in metal interconnects when a high electrical current density is applied. If the interconnect line is not sufficiently confined, the diffusion process may continue until a void forms at the cathode end, eventually leading to failure of the line. If the line is confined, the mass transport due to electromigration eventually leads to a build-up of a stress gradient with a diffusion driving force that is equal and opposite to that due to the high current density, and the mass transport is arrested. The push for increased performance and continued miniaturization in microelectronic devices leads to higher current densities that are more likely to cause electromigration induced failure. Understanding and being able to model this phenomena is important for electronic package design. Modeling and simulation coupled with X-ray microbeam studies, where local elastic strains are measured, can be used to resolve some of the challenging questions regarding the physics of electromigration. This talk provides an introduction to the prevalent electromigration modeling methodology and key issues that are currently unresolved. A finite element modeling formulation is presented and used with two X-ray microbeam data sets, exhibiting opposing trends, to demonstrate how a combination of modeling and simulation may be used to address some of these issues.

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Measuring the Stress Field Around an Evolving Crack in Tensile Deformed Mg AZ31 Using 3DXRD Grain Center Mapping

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(Co-Authors: B. Camin, S. Schmidt, L.P. Mikkelsen, H.O. Sørensen, U. Lienert, H.F. Poulsen and W. Reimers)

The three-dimensional stress field evolving around a notch in a coarse-grained Mg AZ31 sample upon tensile loading has been studied using the grain center mapping variant of three-dimensional X-ray diffraction (3DXRD) microscopy. Notably grain centre mapping enables in situ measurements of the stress field around local phenomena such as crack tips in coarse-grained specimens where the conventional “powder-like” scanning approaches for fine-grained samples do not apply. The technique gives grain resolved centre-of-mass positions, orientations and stress tensors, and using the individual grains as probes 3D information about the maximum stress values and locations, and in particular the extent of the plastically deformed zone around the notch tip, can be obtained. This is of special interest due to the influence on crack growth. In the presentation the technique is introduced, and the obtained results are compared with tomographic evidence of crack growth as well as macroscopic FEM simulations.

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Advances in Understanding Deformation Structures From High Resolution Reciprocal Space Mapping

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(Co-Authors: C. Wejdemann, B. Jakobsen, U. Lienert, and H.F. Poulsen)

With high angular resolution high energy three-dimensional X-ray diffraction established at APS beamline 1-ID quantitative information is gained about dislocation structures in individual grains in the bulk of a macroscopic specimen by acquiring reciprocal space maps. In high resolution three-dimensional reciprocal space maps of tensile deformed copper, individual, almost dislocation-free subgrains are identified from high-intensity peaks and distinguished by their unique combination of orientation and elastic strain; dislocation walls manifest themselves as a smooth cloud of lower intensity. The elastic strain varies only slightly within each subgrain, but significantly between different subgrains. In average, subgrains experience backward strains, whereas dislocations walls are strained in forward direction. Based on these observations a revision of the classical composite model for explaining asymmetric peak profiles is formulated in full accordance with the experimental findings. When pre-deformed specimens are pulled along a perpendicular direction, a reversal of the radial profile asymmetry is observed. The structural evolution during such a strain path change is rationalized in the above mentioned model.

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The need for 3D Data on Elastic and Plastic Response of Polycrystals

A. Rollett, Carn. Mell. Univ., rollett@andrew.cmu.edu

Understanding the response of polycrystalline materials to mechanical loading encompasses a broad range of fundamental issues as well as applications. In many cases, we are able to predict the average elastic or plastic response, although even elastic stiffness is a challenge in complex materials such as bone. However, when one considers properties such as fracture toughness, fatigue resistance, shock loading and so on, our quantitative understanding is less good. In general terms, many useful mechanical properties depend on the tails of distributions of such quantities as grain size, local stress, local accumulated slip and so on. Since polycrystalline materials are self-evidently three-dimensional, synchrotron-based methods such as High Energy Diffraction Microscopy have the attractive feature of providing (potentially) full 3D orientation maps. When supplemented by far-field diffraction experiments, more information should be available for comparison with the many different simulation tools. In particular, finite element and image-based methods provide full-field solutions for mechanical probes of materials. High energy x-ray experiments appear to be the best available tool for validating such computations.

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Status and Future opportunities of 3DXRD Software

S. Schmidt, Risø /DTU, ssch@risoe.dtu.dk

The 3DXRD (Three Dimensional X-ray Diffraction) methodology facilitates non-destructive characterization of the microstructure in the bulk of materials. The FABLE platform (Fully Automatic BeamLines and Experiments) is a collection of data analysis and simulation programs for 3DXRD investigation of polycrystalline materials. The talk will give an overview of current FABLE software along with future activities.

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FABLE

S. Schmidt, Risø /DTU, ssch@risoe.dtu.dk

(Co-Authors: J. Wright, J. Oddershede and H. Sørensen)

(Saturday Lecture) The FABLE software platform comprises a suite of programs for analysis of 3DXRD diffraction data. For polycrystalline materials information such as crystallographic orientation, position, volume, shape and strain can be obtained on the individual grains. The session will give an overview of the various algorithms/programs including an online demonstration of a typical data analysis procedure.

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In Situ Studies of Engineering Processes With Synchrotron Radiation

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High-energy X-rays offer the large penetration depths that often are required for the determination of bulk properties in engineering materials research. In addition, new sources provide very high intensities on the sample which can be used not only for high spatial resolution using very small beams, but also for high time resolution in combination with a fast detector. This opens up possibilities for a wide range of engineering specific in situ experiments. Typical examples that are already widely used are heating or tensile testing in the beam. However, there are also more challenging in situ experiments in the field of engineering materials research like e.g. friction stir welding, dilatometry, or cutting. Selected examples of such experiments will be presented.

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Building Lattice Strain Pole Figures Using Aggregate-Scale High Energy Diffraction Experiments

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A new generation of material probes using high energy synchrotron x-rays are being developed to understand deformation partitioning in polycrystals. In a powder or aggregate experiment, a diffraction volume containing many thousand crystals is interrogated in transmission producing full Debye rings on the detector. By rotating the sample, all crystal orientations within the aggregate are interrogated. Using the fundamentals from Quantitative Texture Analysis, lattice strains from the aggregate experiments can be depicted on lattice Strain Pole Figures (SPFs) for various families of lattice planes. A representative diffraction volume can be constructed by matching the distribution of orientations measured in the physical sample. This model aggregate can be loaded using crystal-based simulation methods and virtual diffraction experiments used to create model SPFs. In this talk, results from in situ cyclic loading experiments on AA 7075-T6 are presented along with crystal-based finite element simulation results. Uncertainty analysis of both experiment and simulation are discussed within the context of the subtle changes that are expected to occur during cyclic loading. A new SPF-based method for measuring residual stress fields is briefly introduced that uses a finite element discretization of the workpiece for imposition of equilibrium and boundary conditions. Residual stress fields in a Low Solvus High Refractory (LSHR) nickel-base superalloy are presented.

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Using 3DXRD and Tomography to Study the Microstructure of Chalk, a Natural Porous Rock

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Determination, by conventional methods, of mineral type, crystal orientation and spatial position of micrometer sized crystals that are embedded in a rock or porous material has been challenging. Traditionally, individual grains must be picked out and analyzed separately. Disintegrating a sample annihilates any possibility for gathering information about the mineral assemblage or the textural relationships within the material. X-ray diffraction (3DXRD), which was pioneered by H. F. Poulsen and coworkers has the potential to also be applied successfully to rocks, soils and sediments. We used a combination of X-ray microtomography (XMT) and 3DXRD to examine samples of very fine-grained chalk and the minerals present in fractures. This study is the first application of 3DXRD on a natural, porous, multiphase material. XMT allows three-dimensional imaging of particles and pore structure at high resolution on samples less than 500 μm in diameter. We used data with voxel size (volume pixel) of 350 nm. The contrast in XMT images is derived from the variation in linear absorption coefficients for the constituting materials. For complex materials, containing unknown phases, the data can be difficult to interpret. For our studies of the flow properties in chalk, it was important to know if the minerals found in the fractures were original, or introduced by drilling. With standard powder X-ray diffraction (XRD) and Mössbauer spectroscopy, we could identify some of the minor phases, but these samples are large compared to the tomography sample and they do not offer spatial information, i.e. it is not possible to tell whether these phases are present in the fractures or elsewhere in the sample. To determine the minor crystalline phases and their position within the sample we employed the method 3DXRD microscopy. The chalk fragment we investigated is composed of nanoscale calcite crystals with a random orientation. In our 3DXRD experiment, these produced powder rings without texture. Superimposed on this pattern, Bragg diffraction peaks resulting from the other crystalline phases could be observed. From these peaks, we could identify crystals of barite and a bit of pyrite. Magnetite, celestite and siderite, other minerals that might have been present, were not observed. We also identified calcite and quartz crystals and defined their positions with reasonable precision.

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Opportunities and Challenges in Orientation Mapping with High Energy Diffraction Microscopy

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We have demonstrated, using data collected at APS 1-ID, the ability to reconstruct crystallographic orientation fields in polycrystals with both well ordered and defected grains. Forward modeling based reconstructions from diffraction images yield micron scale resolution digital representations of cubic millimeter volumes of material with of order 0.1 degree orientation resolution. It is relatively straightforward to combine HEDM data sets with absorption tomography in order to track, for example, sample shape change under strain or the presence of voids. Brief illustrations will be given of measurements of grain growth in pure nickel and ductile straining of a copper sample. Since the measurements are non-destructive, we follow the evolution of ensembles of grains as they respond to treatments. This experimental ability is making possible direct comparisons between observed evolution and model based computations that take measured structures as input. The range of materials issues that can potentially be addressed by this and similar approaches is very large. The list of interested parties is growing rapidly. Beam time and experienced personnel are, of course, limited. Further, we are just learning how to deal with the large output data sets and to track features between successive states. As with any new measurement technique, one has to learn about which algorithms/features/trends are robust versus tenuous. In some cases, the uniqueness of the measurement makes independent verification difficult or impossible. These are all growing pains that need to be addressed by the community as well as by individual groups.

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Combining Grain Boundary Tracking and XRD for 3-Dimensional Grain Mapping of Deformed Polycrystalline Al-alloy

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A novel method that can provide accurate analysis of individual grains has been produced by combining grain boundary tracking (GBT) with X-Ray diffraction (XRD) microscopy. Since XRD is a non-destructive technique for characterizing bulk materials, in-situ analysis of metals can be applied close to the point of fracture whilst still producing practical data. This combination of techniques, given the acronym GBTXRD, provides accurate information about individual grain orientations from near field XRD analysis, whilst the grain boundary tracking accesses 1 micron level analysis of grain morphologies in 3-dimensions. XRD was performed using an X-ray pencil beam was employed to analysis a specimen of Al-3mass% Cu before and after deformation. The morphologies of the grains were then determined from computer tomography (CT) imaging of the deformed specimen after being subjected to a liquid metal wetting method using Ga. Grain boundary tracking method was then applied to the CT images to provided an accurate description of the position and morphology of the grains. A system has been developed to determining which diffraction spots were related to which grain, by grouping the beams position and the associated diffraction spots. From this data it was possible to describe the grain misorientation.

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Studying Local Strains in Aggregates Via Modeling and Diffraction Techniques

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Polycrystal plasticity models are used for simulating and interpreting in-situ internal strain measurements done with neutron and X-ray diffraction. Experimental information, on the other hand, allows us to characterize deformation mechanisms responsible for plasticity at the lower scale level. We use either an Effective Medium or a Local Field model for simulating aggregate response. The former is an Elasto-Plastic Self-Consistent (EPSC) model [1] which allows stress and strain to differ from grain to grain depending on their directional anisotropies, but enforces homogeneity of stress and strain in the grain domain. The latter is an Elasto-Visco-Plastic (EVP-FFT) model [2], based on Fast Fourier Transform for solving the local stress equilibrium equation. We use in-situ diffraction measurements, performed on Mg aggregates at SMARTS-LANSCE [3], the APS 1-ID beam [4], and the 34-ID beam, to provide information to models for understanding the role that twinning plays in deformation. We measure, specifically, the effect of twin growth upon average internal stress re-accommodation inside the parent grain, inside the twin, and inside neighboring grains. We also measure local stresses in the vicinity of a twin. We discuss how much of these experimental evidence can be incorporated into models, how much of it can be explained through modeling, and how models can help us elucidate nano-scale mechanism at the grain level.

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Aircraft Engine Reliability: How High Energy X-rays Can Help

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Over the ~65 year history of jet powered commercial air travel, the performance, durability and reliability of jet engines has improved with each new generation. In 1958, when the Boeing 707 entered commercial revenue service, the goal was for the engines to operate for 500 flight hours between overhaul. Today long haul aircraft such as the Boeing 747 or Airbus 340 use engines that remain on wing for more than 20,000 hours. This improvement is possible due to a variety of factors ranging from better designs to improved materials. This talk will describe some of these improvements including the refinements in calculating the life of critical components. It is the extended life of these components that has enabled longer operating times without increased risk of in-flight failure. Even so, many expensive critical components are retired having considerable remaining useful life because of the uncertainty of current life estimates. One of the next steps in refining these life calculations will involve improved means for incorporating the role of residual stresses. The use of high energy x-rays is a critical factor in measuring residual stress with better accuracy and in achieving this improvement. This also will be discussed.

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Interaction Between Grain Center Mapping by 3DXRD and Polycrystalline Deformation Modelling

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To advance our scientific understanding of polycrystalline deformation beyond the first exciting observations, careful thinking on how to best match experiment types, data analysis and modelling approaches is called for. Interaction between experiment and modelling in the initial planning phase is important to match the deformation conditions, the data resolution (spatial, temporal and angular) as well as the measured quantities of the experiment to the material selected and the materials science problem to be investigated. Grain center mapping has the advantage of measuring lattice rotations for a sufficiently large number of individual grains to draw statistically valid conclusions. On-going projects will be briefly presented with emphasis on how to exploit the interaction between experiments and modelling, often by means of data analysis to identify the critical parameters. A series of grain centre maps measured at different strain levels are obviously ideally suited to evaluate models operating with the grain as the basic entity defined by its crystallographic orientation, i.e. the Taylor model and its derivatives. The large number of grains covered in a map allows analysis of the relative effects of the crystallographic orientation and other parameters. The ability to combine the lattice rotation with the full three-dimensional elastic strain tensor of the grain provides specific information on the interaction of the grain with its neighbors. Such data may be analyzed considering only the grain itself, in terms of resolved shear stresses and therefore the identity of the activated slip systems during plastic deformation. Statistical analysis of the entire ensemble of grains may also provide information about the behavior of subsets of grains with specific characteristics (similar orientation, elastic compliance or Taylor factor, for example). The possibility of generating approximate space-filling maps based on grain centre maps further opens up for comparison with and further development of detailed models for grain interaction, e.g. based on finite element or Fourier approaches. The mesh of such models is typically smaller than the grain size, i.e. also modelling intra-granular phenomena, which may be subsequently averaged over the grain. Grain centre maps may therefore also improve our understanding of processes at scales smaller than the probed grain scale. Grain center maps may be combined with data from other types of experiments probing intragranular phenomena.

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Microstructure Modeling of Irradiated Materials

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The critical ingredient needed for the development of a predictive, materials-physics based modeling approach for materials under irradiation is a better understanding of microstructural processes. However, in spite of the well-known existence of microstructural phenomena in nuclear materials, such as void swelling, fission-gas release and crack development, a comprehensive understanding of materials under irradiation remains to be developed. This lecture will provide a brief overview and vision on how such a capability can be developed by an atomistically-informed mesoscale modeling approach. The critical need for experiments will be emphasized, not only for providing key input parameters to the simulations but also for the validation of key predictions. This work was supported by U. Chicago Argonne, LLC, Operator of Argonne National Laboratory, a U.S. Department of Energy Office of Science laboratory operated under Contract No. DE-AC02-06CH11357.

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Recent Developments at Beamline ID11 at the ESRF

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The 3DXRD microscope has been in regular operation at the Materials Science beamline ID11 at the ESRF since the year 2000. The instrument uses high X-ray energies and microfocus beams to perform in-situ and non-destructive bulk measurements in 3D by using X-ray diffraction. Since the inception of the instrument, continuous developments have been in progress to improve the spatial and temporal resolution of the instrument and also to make the technique both more user friendly and applicable to a wider range of samples. Currently a new diffractometer is being installed as 3DXRD station which offers submicron positional accuracy and has tilt stages to enable topo-tomography data collections to be carried out. This is combined with a 3D detector that has two high resolution phosphor screens and can be used for mapping deformed grain structures. Recent additions to the beamline include dedicated emplacements for grain tracking and tomography; and also a nanoscope station which offers X-ray beamsizes of less than 200 nm at 42 keV.